

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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In re Patent Application of:  
Kouichi KITAHATA et al.

Application No.: 10/571,582

Confirmation No.: 9795

Filed: March 10, 2006

Art Unit: 1611

For: POROUS SILICA HAVING SUBSTANCE  
CARRIED THEREON

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Examiner: ORWIG, Kevin S.

DECLARATION UNDER 37 CFR 1.132

COMMISSIONER FOR PATENTS  
P. O. Box 1450  
Alexandria, VA 22313-1450

Sir:

I, Kouichi KITAHATA, residing in Mic-ken, Japan, hereby declares and states as follows:

1. That I am a co-inventor of the above-identified application, and thoroughly familiar with the contents of U.S. Application Serial No. 10/571,582 filed on March 10, 2006, entitled POROUS SILICA HAVING SUBSTANCE CARRIED THEREON, its prosecution before the United States Patent and Trademark Office and the references cited therin.

2. I am a graduate of The Kagoshima University, Faculty of Agriculture and received a master's degree in the year 1995, majoring in Biochemical Science and Technology.

3. That I have been employed in Taiyo Kagaku Co., Ltd. in the year 1996 and have been assigned to the Research Laboratories.

4. I have been involved in the research and development of mesoporous silica since 2003.

5. The following experiments were conducted by myself or under my direct supervision and control in order to compare and study the porous silica of the present invention with the mesoporous silica of Shio (U.S. Patent No. 6,511,668), particularly in their structures.

METHODS

(i) Appendix I(a copy of FIG. 14 of Shio) shows a nitrogen adsorption isotherm diagram of the rod-like mesoporous powder obtained in Shio.

Also, a nitrogen adsorption isotherm diagram was obtained for the porous silica obtained in Preparation Example 1 of the present specification using a water glass as a raw material. Specifically, the nitrogen adsorption isotherm diagram was obtained using a nitrogen adsorption analyzing instrument "Quadasorb" SI (commercially available from Quantachrome Instruments).

## RESULTS

The results are shown in FIG. I.

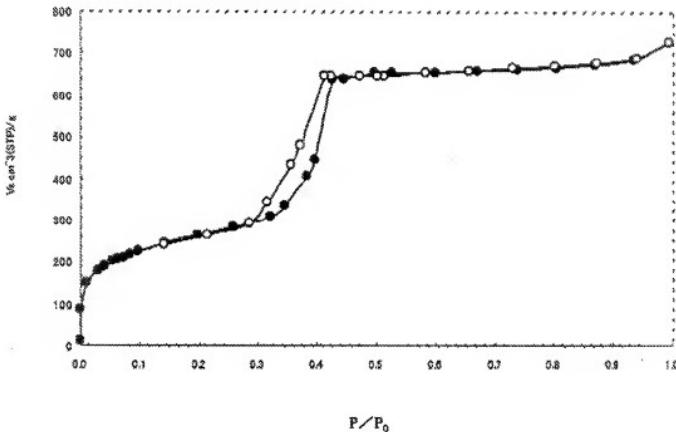


FIG. I

## DISCUSSION

Since the same mesoporous silica as that disclosed in Shio could not be made available, I studied the nitrogen adsorption isotherm diagrams of the porous silica of the present invention and the mesoporous silica of Shio to clarify the structural differences therebetween.

When the nitrogen adsorption isotherm diagram obtained for the porous silica of the present invention is compared with that of the mesoporous silica of Shio, in a region

satisfying  $P/P_0$  of 0.8 or greater, a curve for the rod-like mesoporous powder of Shio shows a dramatic elevation in  $V_a$ , whereas a curve for the porous silica of the present invention shows hardly any increase thereof. It is confirmed from the difference between the diagrams that the rod-like mesoporous powder disclosed in Shio contains a large number of macro-pores of about 100 nm or more located externally in the gaps between the powders, and such pores hardly exist in the porous silica of the present invention. It is considered that such a considerable difference in the structures of both of the porous silicas are incurred by the difference in the raw materials used. Specifically, the raw material of the porous silica of the present invention is water glass, where an  $\text{SiO}_2/\text{Y}_2\text{O}$  ratio is 2 or more. By contrast, the raw material for the rod-like mesoporous silica in Shio is a silicate that has an  $\text{SiO}_2/\text{Y}_2\text{O}$  ratio satisfying  $0 < \text{SiO}_2/\text{Y}_2\text{O} < 2$ . This matter is described in the phrase "a rod-like mesoporous powder or a rod-like non-porous powder can be prepared by prescribing the concentration of the silicate within the specific range" described on column 4, lines 28 to 57 of Shio, especially lines 55 to 57.

Moreover, in general, when perfume is added to a rod-like mesoporous silica powder at a certain level, the presence of the large number of macro-pores in the gap between the mesoporous silica particles causes weak adsorption in the gap or space. And since the macro-pores hold markedly larger space as compared to the size of the perfume molecules, the adsorption is very weak, in fact, so weak that sufficient sustained-release effects cannot be obtained after the adsorption. Therefore, it can be seen that in order to obtain sufficient sustained-release effects, a perfume such as menthol must be supported within the pores of the porous silica as described in the present invention.

**Statement Under 18 U.S.C. § 1001**

The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

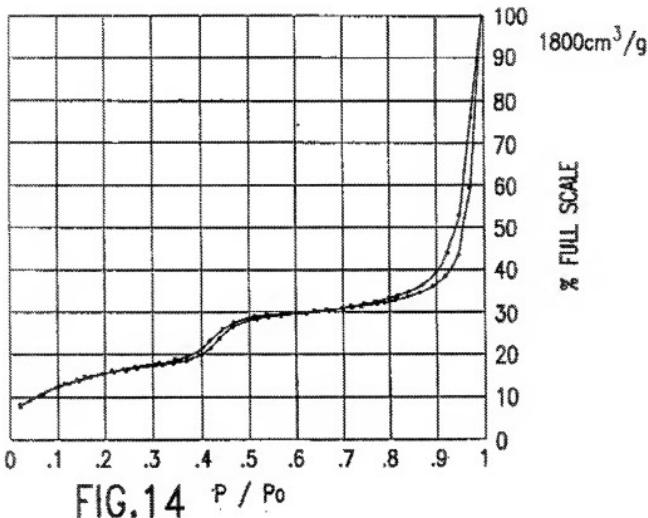
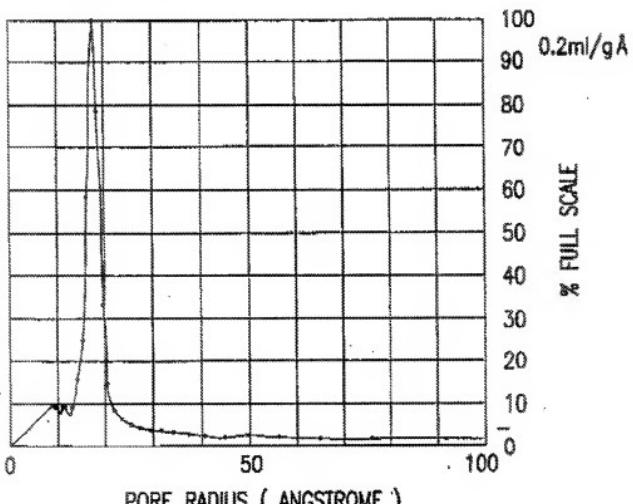
Kouichi KITAHATA

Kouichi KITAHATA

October 30, 2009

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Daté

FIG.14  $P / P_0$ 

PORE RADIUS ( ANGSTROME )

FIG.15